REPORT ON MICROCHEMICAL METHODS

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There has been no report on microchemical methods since the one made in 1941 by the late E. P. Clark. Inasmuch as the recognition of microanalytical methods has steadily increased it is desirable that referee work leading to the standardization of these methods be resumed.

Since about forty quantitative microchemical methods are now in use, it seemed too arbitrary for the Referees to select the methods for study or to designate the methods to be studied first. Few, if any, of the laboratories equipped to perform microanalyses employ all the micro methods. It was not known what determinations each laboratory was making or was prepared to make, nor was it known which laboratories would be willing to participate in collaborative studies on microchemical methods. Consequently, a questionnaire was sent to microchemists throughout the country asking for their opinions on the following questions: (1) Should standardization of microchemical methods be attempted? (2) Which determinations should be standardized? (3) Which determinations should receive first attention? Also in the questionnaire were the questions: (1) Would you participate in collaborative studies? (2) What determinations are you performing in your laboratory?

More than seventy-three replies to the questionnaire have been received, and returns are still not complete. The replies have clearly shown that collaborative work on standardization of micro methods is endorsed by the microanalysts throughout the country. Of the seventy-three microchemists who replied, none were opposed to the studies, and only three were indifferent. Many not only answered the questionnaire but also endorsed the proposed work. The response to the question regarding willingness to cooperate in these studies has also been gratifying. Forty-seven expressed a desire to participate, and most of the twenty-six unable to collaborate stated that they were not able to do so because of the nature of their present work or lack of equipment or personnel.

To determine the geographic location of the microanalytical laboratories in this country, the replies received from the East, Midwest, and West were tabulated, with the following results: 70 per cent are in the East, 20 per cent in the Midwest, and 10 per cent on the West Coast.

The replies to the question regarding methods that should be standardized mentioned eighteen in addition to the eighteen listed in the questionnaire, making a total of thirty-six suggested. The ten determinations that

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received the most votes for standardization were: Carbon and hydrogen (37), sulfur (37), Kjeldahl nitrogen (36), molecular weight (36), Dumas nitrogen (35), chlorine (30), bromine (27), iodine (26), acyl group (25), alkoxyl group (25). These were closely followed by phosphorus, hydroxyl group, neutralization equivalent, and oxygen. The remainder received 18 or fewer votes. A large percentage of the chemists who regularly perform a determination recommended standardization of the method.

The selections of methods to be studied first, second, third, and fourth were weighted on a point system by multiplying each first choice by 4, second by 3, third by 2, and fourth by 1. The ten determinations which received the largest number of points were: Carbon and hydrogen (114), Dumas nitrogen (69), Kjeldahl nitrogen (56), sulfur (39), chlorine (33), molecular weight (31), oxygen (19), bromine (13), iodine (12), and acyl groups (11).

In view of these replies to the questionnaire, the Referee and Associate Referee recommend† that collaborative studies be initiated on the methods for the determination of carbon and hydrogen, and the Dumas and Kjeldahl methods, for the determination of nitrogen.

† For report of Subcommittee C and action of the Association, see *This Journal*, 31, 53 (1948).